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(71) Applicant (*for all designated States except US*): STEPAN COMPANY [US/US]; 22 West Frontage Road, Northfield, IL 60093 (US).

(72) Inventor; and

(75) Inventor/Applicant (*for US only*): FAUNCE, James, A. [US/US]; 705 Pinehurst, North Aurora, IL 60542 (US).

(74) Agent: SCHARFF, Christopher, M.; McAndrews, Held & Malloy, Ltd., 500 West Madison Street, 34th Floor, Chicago, IL 60661 (US).

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(54) Title: HYDROLYTICALLY STABLE PHTHALATE ESTER LUBRICANTS AND METHOD OF METAL WORKING WITH HYDROLYTICALLY STABLE PHTHALATE ESTERS LUBRICANTS

(57) Abstract: A water-based lubricant is provided that includes a phthalate ester in an amount of about 2 % to about 20 % based on the weight of the final composition and water in an amount of about 60 % to about 93 % based on the weight of the final composition. Also provided are a method of making the water-based lubricant and a method of metalworking using the water-based lubricant.

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HYDROLYTICALLY STABLE PHTHALATE ESTER LUBRICANTS AND METHOD  
OF METALWORKING WITH HYDROLYTICALLY STABLE PHTHALATE ESTERS  
LUBRICANTS

RELATED APPLICATIONS

[0001] This Application claims priority to U.S. Provisional Application No. 60/435,071, filed December 20, 2002.

BACKGROUND OF THE INVENTION

[0002] The metalworking industry requires lubricants in many of its operations. Water-based lubricants are particularly desirable because of the ease of using and disposing of the water base. Adipate esters have also been used as successful lubricants in the metalworking industry. Adipate esters, however, are unstable in water solutions and thus less desirable than water-based lubricants. In the past, it has been determined that esters having groups with steric bulk immediately adjacent the ester group may lend some hydrolytic stability to lubricants. The metalworking industry has gone to utilizing isopropyl and 2-ethylhexyl esters as a source of hydrolytic stability due to the steric bulk of the groups adjacent to the ester linkage. However, there continues to be a need for hydrolytically stable lubricants for use in the metalworking industry.

BRIEF SUMMARY OF THE INVENTION

[0003] The present invention provides a hydrolytically stable phthalate ester lubricant and a method of metalworking with a hydrolytically stable phthalate ester lubricant. The phthalate esters used in the present invention have shown superior hydrolytic stability versus other esters and have shown successful lubrication results in ASTM testing for lubricants.

## DETAILED DESCRIPTION OF THE INVENTION

[0004] The phthalate esters that may be used in the lubricant of the present invention include (I) a phthalate polyester-ether polyol, (II) a phthalic anhydride reacted with an equivalent of a fatty alcohol which is then ethoxylated with a variety of moles of ethylene oxide (or propylene oxide), and (III) the amine-neutralized salts of item (II).

[0005] Referring to (I), the phthalate polyester-ether polyol is the reaction product of:

(1) about 2 - 60 % based on the weight of the polyester-ether polyol of phthalic anhydride or phthalic acid;

(2) about 40 - 98 % based on the weight of the polyester-ether polyol of at least one polyol of the formula:

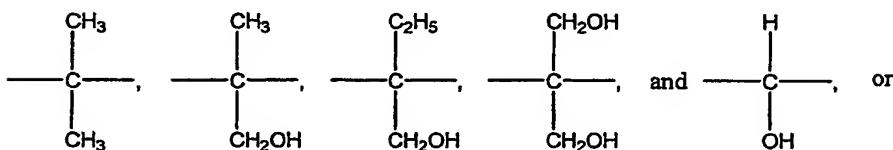


wherein  $\text{R}_1$  represents:

(a) alkylene groups of about 2 to 10 carbon atoms;

(b)  $-\text{CH}_2-\text{R}_2-\text{CH}_2-$

where  $\text{R}_2$  represents:



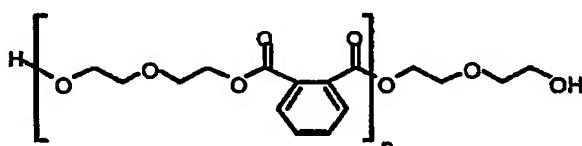
(c)  $-(\text{R}_3\text{O})_n-\text{R}_3-$

where each  $\text{R}_3$  independently is an alkylene group of about 2 to about 4 carbon atoms, and  $n$  is an integer of from about 1 - 200; and

(3) about 10 - 80 % based on the weight of the polyester-ether polyol of an alkoxylating agent.

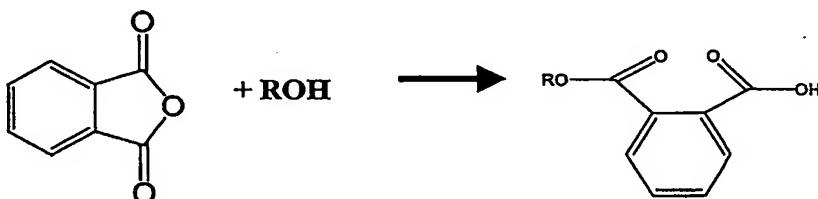
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[0006] A preferred phthalate polyester-ether polyol is a propoxylated diethylene glycol-phthalic anhydride-based polyester polyol. The diethylene glycol-phthalic anhydride-based polyester polyol is sold by the Stepan Company under the tradename Stepanpol PS-2002. Stepanpol PS-2002 has the following structural formula.



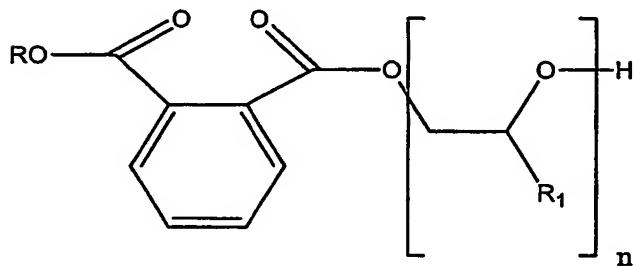
(n ~ 2.0)

Regarding (II), the phthalic anhydride - fatty alcohol reaction is described as follows.



[0007] where R is C<sub>4</sub> to C<sub>22</sub>, branched or linear.

[0008] The ethoxylation reaction of the product of the phthalic anhydride - fatty acid reaction proceeds by ethoxylating the product with a variety of moles of ethylene oxide (or propylene oxide or butylene oxide) resulting in the following structure.



where R<sub>1</sub> = H, CH<sub>3</sub>, CH<sub>2</sub>CH<sub>3</sub>, and n = 1-20, preferably 1-10.

[0009] Regarding (III), the amine-neutralized salts of (II) can also be used as the phthalate ester in the present invention. The amines that may be used to accomplish this

neutralization include triethanolamine, triethylamine, triethanolamine, monoethanolamine, 2-ethylhexylamine, tallow amine ethoxylates or any other amine in general.

[0010] The stability of the phthalate esters of the present invention was investigated in both acidic and basic conditions and compared to adipic ester, a known lubricant often used in the metalworking industry.

[0011] First, testing results of the hydrolytic stability of the propoxylated Stepanpol PS-2002 compared with adipic ester in an acidic system confirmed that the phthalate esters of the present invention provide the hydrolytic stability in acidic conditions desirable for lubricants in the metalworking industry. Specifically, the propoxylated Stepanpol PS-2002 did not exhibit the ester breakdown, and subsequent acid value increase, of the adipic ester.

[0012] Additionally, tests were run which compared the hydrolytic stability of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in a basic system. Base stability can be observed by measuring the breakdown in molecular weight of the ester over time. The breakdown in molecular weight via gel permeation chromatography (GPC) of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in 0.50M KOH and 0.50M TEA (triethyl amine) were calculated by measuring the area percent of the highest molecular weight species in a GPC graph. Testing revealed that the area percent of the adipate polyol decreased drastically over time. In contrast, the area percent of the propoxylated Stepanpol PS-2002 did not decrease over the same time period.

[0013] Third, tests were run to illustrate the hydrolytic stability of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in a KOH system. The average molecular weight of the systems were directly measured to demonstrate the respective stability in a basic system. The average molecular weight of the adipate polyol decreased drastically over time. In contrast, the average molecular weight of the propoxylated Stepanpol PS-2002 did not decrease over the same time period, confirming the hydrolytic stability of the phthalate ester.

[0014] Accordingly, the phthalate esters of the present invention provide the hydrolytic stability in basic conditions desirable for lubricants in the metalworking industry.

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[0015] The lubricant of the present invention was compared to metalworking industry lubricant standards (isopropyl oleate and 2-ethylhexyl oleate). In particular, the tests run were a 4-ball wear, Extreme Pressure Pin and Vee, and a Tapping Torque. All tests were run via an ASTM method.

[0016] Table I sets forth the results for the ASTM D-4172 4-ball wear testing.

TABLE I

Product	Wear Diameter (mm)
Stepan MWA-560 HS	0.598
2-ethylhexyl oleate	0.752
isopropyl oleate	0.628

[0017] Table II sets forth the results for the ASTM D-3233 Extreme Pressure Pin and Vee testing.

TABLE II

Product	Failure Load (lbs)
Stepan MWA-560 HS	1000
2-ethylhexyl oleate	1250
isopropyl oleate	1250

[0018] Table III sets forth the results for the ASTM D-5619 Tapping Torque testing.

TABLE III

Product	Efficiency (%)
Stepan MWA-560 HS	96.20
isopropyl oleate	110.28

[0019] The results as set forth in Tables I, II, and III demonstrate that the lubricants of the present invention provide lubricating properties comparable to those of the metalworking

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industry standards. As such, the lubricants of the present invention may be utilized in any application in the metalworking industry that requires a lubricant such as isopropyloleate or 2-ethylhexyl oleate.

[0020] A typical lubricant formulation useful in the metalworking industry may consist of up to about 90% water, about 5% phthalate ester, and about 5% triethanolamine. Those skilled in the art recognize that many lubricant formulations are maintained as proprietary trade secret information, and that the phthalate esters disclosed above may be utilized as the main lubricating ingredient in those proprietary lubricant formulations. Thus, the present invention includes such lubricant formulations that utilize the phthalate ester as the main lubricating ingredient. In other words, a person skilled in the art may take his or her proprietary formulation and in the place of the prior main lubricating ingredient utilize the phthalate esters to arrive with a lubricant formulation of the present invention. Such proprietary formulation usually include water, the main lubricating ingredient and at least one other ingredient. The at least one other ingredient may be a single ingredient, as described in the formulation above where the at least one other ingredient is triethanolamine, or may include any number of components.

[0021] Typical water-based lubricants presently include water in an amount between about 60% and about 93% by weight of the total composition, preferably about 75 to about 87%, and a main lubricating ingredient in an amount between about 2% and about 20% by weight of the total composition, preferably about 5 to about 10%. The phthalate esters disclosed above may be used as the main lubricating ingredient in such water-based lubricants and in such amounts. Typical water-based lubricants presently further include at least one other ingredient in a total weight of about 2% and about 20% based on the final composition. One typical component of the at least one other ingredients is an amine in an amount of between about 2% and about 10% by weight of the total composition. The amine is typically used to regulate the pH of the lubricant. Thus, the phthalate esters disclosed above can be used in presently used lubricants by replacing the main lubricating ingredient in those present day lubricants with the disclosed phthalate esters to provide the lubricant of the present invention.

APPENDIX

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[0022] The phthalate esters disclosed above may also be used as the main lubricating ingredient in such water-based lubricants including this at least one other ingredient.

[0023] The invention has been described with reference to preferred and alternate embodiments. Modifications and alterations will occur to others upon the reading and understanding of the specification. It is intended to include all such modifications and alterations insofar as they come within the scope of the appended claims or equivalents thereof.